National Reference Laboratory Feed Additives – Control and Authorisation

End of Year Report
2019 - 2020

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1. Introduction

Regulation (EC) No. 882/2004 *on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules* established a network of European and National Reference laboratories. Regulation (EC) No 882/2004 was repealed with effect from 14 December 2019 and replaced by Regulation (EU) 2017/625 *on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products*. Regulation 2017/625 supplements Regulation (EC) No 178/2002 *laying down the general principles and requirements of food law, establishing the European Food Safety Authority and laying down procedures in matters of food safety*, aims for a high level of:

- protection of human, animal and plant health and of the environment via veterinary and phytosanitary measures;
- consumer protection in the internal market; and
- animal welfare along the agri-food chain.

In each area of food and feed control an EU Reference Laboratory (EURL) is identified to coordinate activities in that area. They are supported by a network of National Reference Laboratories (NRLs) which co-ordinate activities within their own member state and contribute to the European wide activities. NRLs are nominated by the Competent Authorities in the respective Member states. In the UK, the Competent Authority for feed additives is the Food Standards Agency (FSA).

The duties of the EURLs and NRLs are set out in legislation however their principal role is to provide analytical and scientific support to ensure that food and feed control is carried out effectively and in a harmonised manner, across the EU member states.

Article 94 of Regulation (EU) 2017/625 describes the responsibilities and tasks of EURLs as follows:

> 1. EURLs shall contribute to the improvement and harmonisation of methods of analysis, test or diagnosis to be used by official laboratories designated in accordance with Article 37(1) and of the analytical, testing and diagnostic data generated by them.
>
> 2. EURLs designated in accordance with Article 93(1) shall be responsible for the following tasks insofar as they are included in the reference laboratories' annual or multiannual work programmes that have been established in conformity with the objectives and priorities of the relevant work programmes adopted by the Commission in accordance with Article 36 of Regulation (EU) No 652/2014:
>
> (a) providing NRLs with details and guidance on the methods of laboratory analysis, testing or diagnosis, including reference methods;
>
> (b) providing reference materials to NRLs;
>
> (c) coordinating the application by the NRLs and, if necessary, by other official laboratories of the methods referred to in point (a), in particular, by organising regular inter- laboratory comparative testing or proficiency tests and by ensuring appropriate follow-up of such comparative testing or proficiency tests in accordance, where available, with internationally accepted protocols, and informing the Commission and the Member States of the results and follow-up to the inter-laboratory comparative testing or proficiency tests;

(d) coordinating practical arrangements necessary to apply new methods of laboratory analysis, testing or diagnosis, and informing NRLs of advances in this field;

(e) conducting training courses for staff from NRLs and, if needed, from other official laboratories, as well as of experts from third countries;

(f) providing scientific and technical assistance to the Commission within the scope of their mission;

(g) providing information on relevant national, Union and international research activities to NRLs;

(h) collaborating within the scope of their mission with laboratories in third countries and with the European Food Safety Authority (EFSA), the European Medicines Agency (EMA) and the European Centre for Disease Prevention and Control (ECDC);

(i) assisting actively in the diagnosis of outbreaks in Member States of foodborne, zoonotic or animal diseases, or of pests of plants, by carrying out confirmatory diagnosis, characterisation and taxonomic or epizootic studies on pathogen isolates or pest specimens;

(j) coordinating or performing tests for the verification of the quality of reagents and lots of reagents used for the diagnosis of foodborne, zoonotic or animal diseases and pests of plants;

(k) where relevant for their area of competence, establishing and maintaining:

(i) reference collections of pests of plants and/or reference strains of pathogenic agents;

(ii) reference collections of materials intended to come into contact with food used to calibrate analytical equipment and provide samples thereof to NRLs

(iii) up-to-date lists of available reference substances and reagents and of manufacturers and suppliers of such substances and reagents; and

(l) where relevant for their area of competence, cooperate among themselves and with the Commission, as appropriate, to develop methods of analysis, testing or diagnosis of high standards.

3. EURLs shall publish the list of the NRLs designated by the Member States in accordance with Article 100(1).

Article 101 of Regulation (EU) 2017/625 describes the responsibilities and tasks of NRLs as follows:

(a) collaborate with the EURLs, and participate in training courses and in inter-laboratory comparative tests organised by these laboratories;

(b) coordinate the activities of official laboratories designated in accordance with Article 37(1) with a view of harmonising and improving the methods of laboratory analysis, test or diagnosis and their use;

(c) where appropriate, organise inter-laboratory comparative testing or proficiency tests between official laboratories, ensure an appropriate follow-up of such tests and inform the competent authorities of the results of such tests and follow-up;

(d) ensure the dissemination to the competent authorities and official laboratories of information that the EURL supplies;

(e) provide within the scope of their mission scientific and technical assistance to the competent authorities for the implementation of MANCPs referred to in Article 109 and of coordinated control programmes adopted in accordance with Article 112;

(f) where relevant, validate the reagents and lots of reagents, establish and maintain up-to-date lists of available reference substances and reagents and of manufacturers and suppliers of such substances and reagents;

(g) where necessary, conduct training courses for the staff of official laboratories designated under Article 37(1); and

(h) assist actively the Member State having designated them in the diagnosis of outbreaks of foodborne, zoonotic or animal diseases or of pests of plants and in case of non-compliance of consignments, by carrying out confirmatory diagnoses, characterisation and epizootic or taxonomic studies on pathogen isolates or pest specimens.

LGC currently holds the NRL roles for feed additives – control and authorisation. Regulation (EC) No. 1831/2003 on additives for use in animal nutrition describes 'feed additives' as substances, micro-organisms or preparations, other than feed material and premixtures, which are intentionally added to feed or water in order to perform, in particular, one or more of the following functions:

(a) favourably affect the characteristics of feed,

(b) favourably affect the characteristics of animal products,

(c) favourably affect the colour of ornamental fish and birds,

(d) satisfy the nutritional needs of animals,

(e) favourably affect the environmental consequences of animal production,

(f) favourably affect animal production, performance or welfare, particularly by affecting the gastro-intestinal flora or digestibility of feedingstuffs, or

(g) have a coccidiostatic or histomonostatic effect.

Feed additives should not:

(a) have an adverse effect on animal health, human health or the environment,

(b) be presented in a manner which may mislead the user,

(c) harm the consumer by impairing the distinctive features of animal products or mislead the consumer with regard to the distinctive features of animal products.

Antibiotics, other than coccidiostats or histomonostats, are not authorised as feed additives.

Depending on their functions and properties feed additives are allocated to one or more of the categories listed in Article 6 of Regulation (EC) No 1831/2003. The categories are:

(a) technological additives: any substance added to feed for a technological purpose;

(b) sensory additives: any substance, the addition of which to feed improves or changes the organoleptic properties of the feed, or the visual characteristics of the food derived from animals;

(c) nutritional additives;

(d) zootechnical additives: any additive used to affect favourably the performance of animals in good health or used to affect favourably the environment;

(e) coccidiostats and histomonostats.

Technological additives, sensory additives, nutritional additives and zootechnical additives have sub-divisions of functional groups to which the feed additives are allocated, as applicable. As a result of technological and scientific development there were various substances that may have a technological effect on feed which was not described in relation to any of the original functional groups. For this reason, in June 2019, a new generic functional group called 'other technological additives' within the category 'technological additives' was created. At the same time a new functional group called 'physiological condition stabilisers' was created in the category 'zootechnical additives'. In addition to good farming practices ensuring the wellbeing of animals and the respect of animal welfare provisions in the EU, scientific studies show that some feed additives may help animals in good health to keep a good physiological condition, to contribute to animal welfare, to favourably affect the animal resilience to stress factors or to support their wellbeing in certain situations. Since the main function of these feed additives could not be allocated to any of the specific functional groups provided for in Regulation (EC) No 1831/2003, it was appropriate to create a new functional group within the category 'zootechnical additives'. The above additions to the functional groups came into force with Commission Regulation (EU) 2019/962. The full list of functional groups within each of the catogories, as of June 2019, are as follows:

1. In the category 'technological additives', the following functional groups are included:

(a) preservatives: substances or, when applicable, micro-organisms which protect feed against deterioration caused by micro-organisms or their metabolites;

(b) antioxidants: substances prolonging the storage life of feedingstuffs and feed materials by protecting them against deterioration caused by oxidation;

(c) emulsifiers: substances that make it possible to form or maintain a homogeneous mixture of two or more immiscible phases in feedingstuffs;

(d) stabilisers: substances which make it possible to maintain the physico- chemical state of feedingstuffs;

(e) thickeners: substances which increase the viscosity of feedingstuffs;

(f) gelling agents: substances which give a feedingstuff texture through the formation of a gel;

(g) binders: substances which increase the tendency of particles of feedingstuffs to adhere;

(h) substances for control of radionucleide contamination: substances that suppress absorption of radionucleides or promote their excretion;

(i) anticaking agents: substances that reduce the tendency of individual particles of a feedingstuff to adhere;

(j) acidity regulators: substances which adjust the pH of feedingstuffs;

(k) silage additives: substances, including enzymes or micro-organisms, intended to be incorporated into feed to improve the production of silage;

(l) denaturants: substances which, when used for the manufacture of processed feedingstuffs, allow the identification of the origin of specific food or feed materials;

(m) substances for reduction of the contamination of feed by mycotoxins: substances that can suppress or reduce the absorption, promote the excretion of mycotoxins or modify their mode of action;

(n) hygiene condition enhancers: substances or, when applicable, microorganisms which favourably affect the hygienic characteristics of feed by reducing a specific microbiological contamination;

(o) other technological additives: substances or, when applicable, microorganisms added to feed for a technological purpose and which favourably affect the characteristics of the feed.

2. In the category 'sensory additives', the following functional groups are included:

(a) colourants:

(i) substances that add or restore colour in feedingstuffs;

(ii) substances which, when fed to animals, add colours to food of animal origin;

(iii) substances which favourably affect the colour of ornamental fish or birds;

(b) flavouring compounds: substances the inclusion of which in feedingstuffs increases feed smell or palatability.

3. In the category 'nutritional additives', the following functional groups are included:

(a) vitamins, pro-vitamins and chemically well-defined substances having similar effect;

(b) compounds of trace elements;

(c) amino acids, their salts and analogues;

(d) urea and its derivatives.

4. In the category 'zootechnical additives', the following functional groups are included:

(a) digestibility enhancers: substances which, when fed to animals, increase the digestibility of the diet, through action on target feed materials;

(b) gut flora stabilisers: micro-organisms or other chemically defined substances, which, when fed to animals, have a positive effect on the gut flora;

(c) substances which favourably affect the environment;

(d) other zootechnical additives;

(e) physiological condition stabilisers: substances or, when applicable microorganisms, which, when fed to animals in good health, favourably affect their physiological condition, including their resilience to stress factors.

Feed additives play an important role in animal nutrition, addressing various aspects such as feed safety, reduction of environmental emissions and sustainability in livestock farming. Before placing feed additives on the market, authorisation must be obtained as specified in Regulation (EC) No 1831/2003. A summary of the authorisation process is given in Appendix 1.

Pursuant to Regulation (EC) No 1831/2003, a list of the currently permitted feed additives can be found in the European Union Register of Feed Additives. The latest edition can be found at:

https://ec.europa.eu/food/safety/animal-feed/feed-additives/eu-register_en (accessed 27 February 2020)

This report provides an update for the National Reference Laboratory role for Feed Additives – Control and Authorisation for the year April 2019 to March 2020.

2. EURL Proficiency Test 2019 – Carotenoids

Carotenoids are classed under the category of sensory additives and are substances which, when fed to animals, add colours to food of animal origin or favourably affect the colour of ornamental fish or birds or add or restore colour in feedingstuffs. Following the training provided in 2018, in 2019 the EURL organised a proficiency test for the determination of the carotenoids astaxanthin, canthaxanthin and adonirubin in samples of poultry feed and fish feed. The invitation to register for the proficiency test (PT) was received on 13 February 2019, with the samples being received on 26 March 2019. The deadline for submission of results was 26 April 2019.

The letter of invitation to participate stated that a limited number of places were available to Official Control Laboratories (OCLs) if they wished to take part (the total number of participants able to take part was stated as being 25). The invitation was forwarded to the UK OCLs, together with a copy of the EURL's method, no responses were received.

The EURL method for the determination of carotenoids involves enzymatic (protease) reaction, acetone extraction and HPLC with detection at 410 nm.

The samples were analysed several times at LGC but there was not sufficient confidence in the PT results to submit them due to the high variability between replicate determinations and the resulting high uncertainty. If this analysis had been carried out as part of an investigation of a formal sample further determinations would have been carried out, however, the limited amount of PT samples provided did not allow for further repeats.

On receipt of the final report z-scores were calculated based on the mean of the results obtained, Table 1. All z-scores were satisfactory, i.e. less than 2.

Analyte	Matrix	Assigned value (mg/kg)	Mean Result (mg/kg)	Z-score (based on mean result)
Astaxanthin	Fish feed	49.965	35.6	-1.1
Canthaxanthin	Fish feed	5.020	2.6	-1.9
Adonirubin	Fish feed	19.365	11.8	-1.6
Canthaxanthin	Poultry feed	10.106	6.4	-1.5

Table 1: Z-scores for carotenoid PT

According to the EURL's report, the assigned values were set as 'the nominal value calculated from the formulation and corrected for the purity of the active substances' which may explain why the majority of results submitted by all participants were less than the assigned values, as a recovery of less than 100 % would be expected.

Figures 1 to 4 show the results and associated uncertainties for all participants in the PT round.

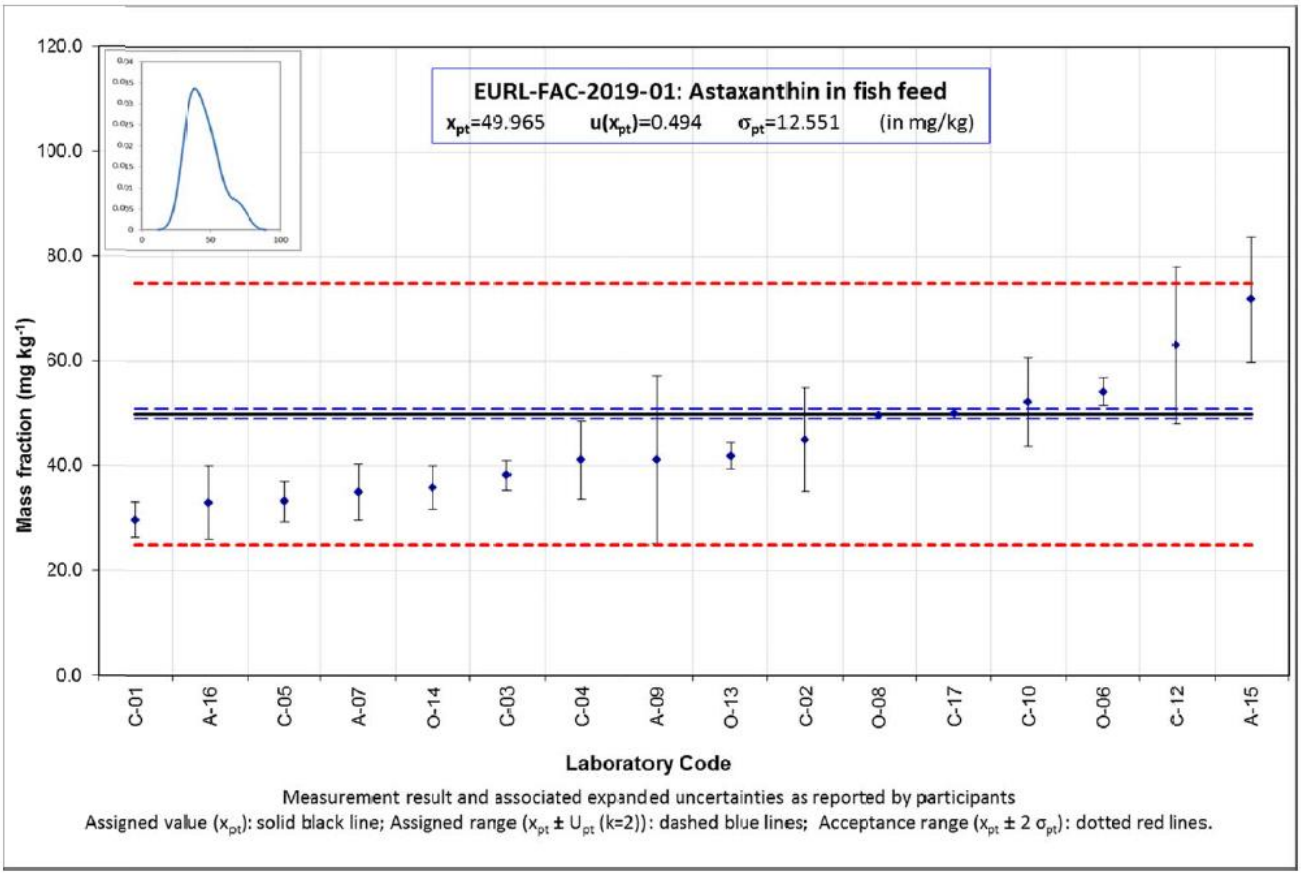

Figure 1: Summary of z-scores obtained by all participants for astaxanthin in fish feed

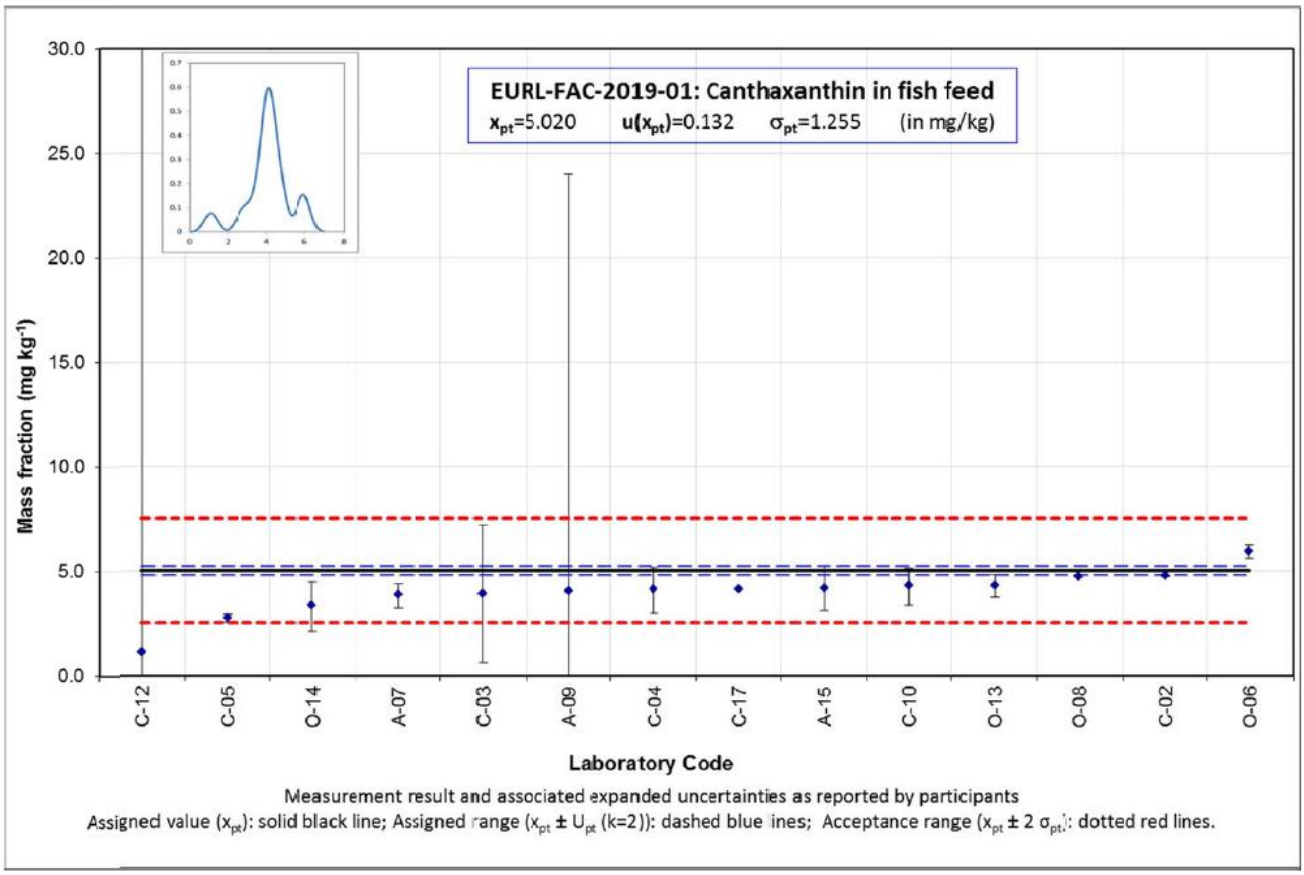

Figure 2: Summary of z-scores obtained by all participants for canthaxanthin in fish feed

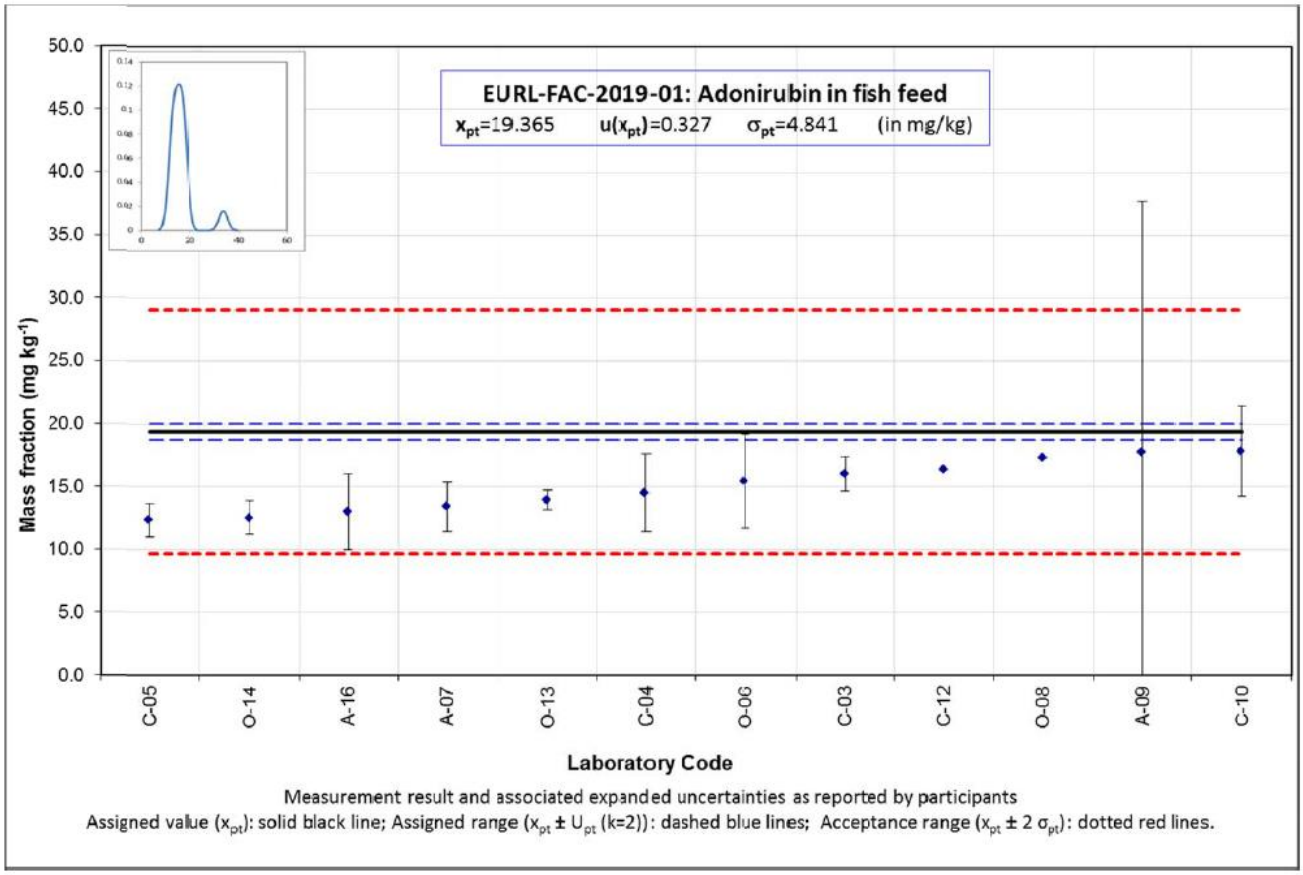

Figure 3: Summary of z-scores obtained by all participants for adonirubin in fish feed

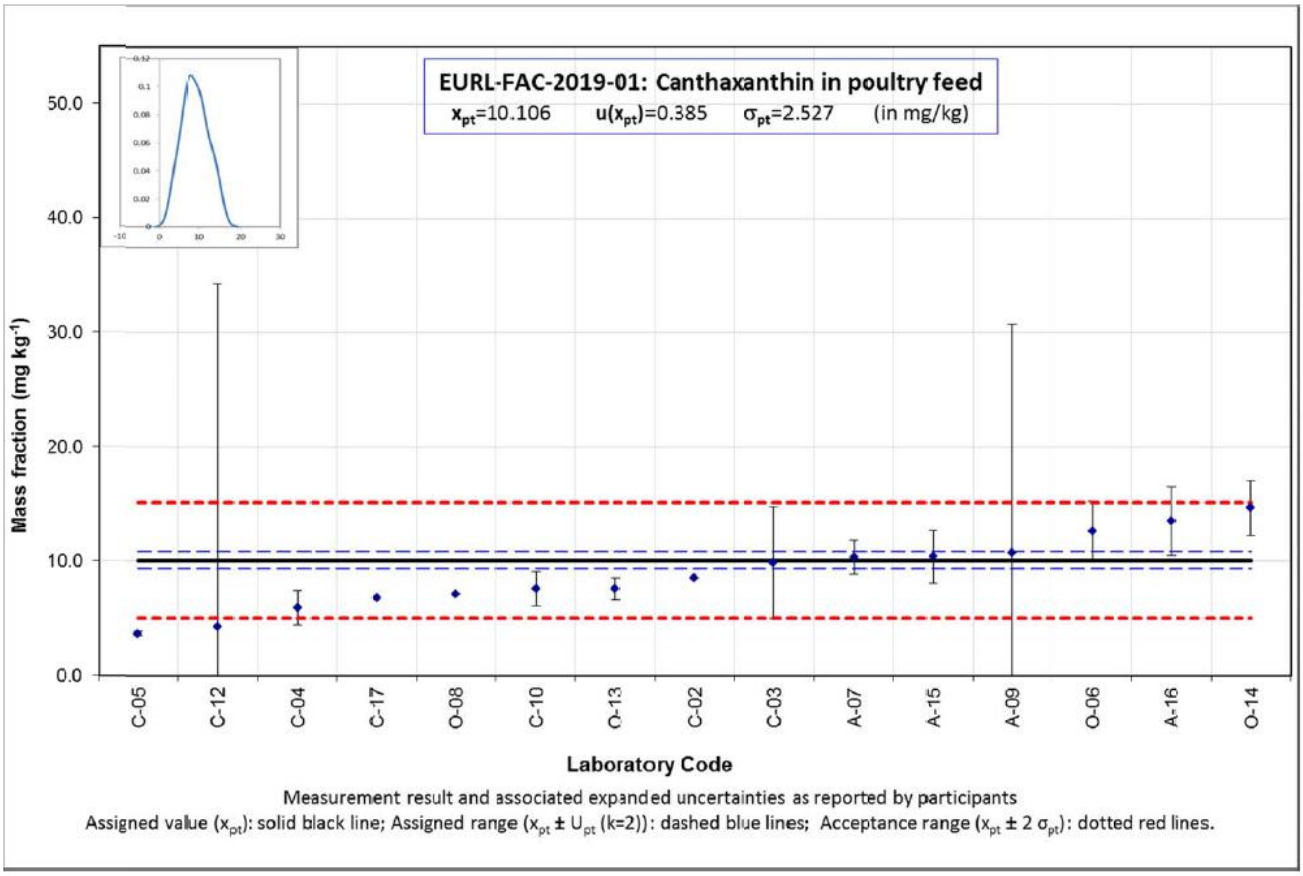

Figure 4: Summary of z-scores obtained by all participants for canthaxanthin in poultry feed

The full report for the proficiency test is not currently available on the European Commission website (https://ec.europa.eu/jrc/en/publications-list) (accessed 8 April 2020).

3. EURL Proficiency Test 2019 - Coccidiostats

In relation to Regulation (EC) No 1831/2003 coccidiostats and histomonostats are defined as substances intended to kill or inhibit protozoa and are the only antibiotics authorised as feed additives.

On 30 April 2019 an invitation to participate in the EURL's second PT of 2019 was received. This focus of the PT was the determination of coccidiostats in compound feed, with the coccidiostats of interest being diclazuril, nicarbazin and narasin. The invitation implied that the coccidiostats could be at authorised or carry-over concentrations. This invitation also stated that the PT was open to OCLs, with a maximum total number of participants of 25. The invitation was forwarded to UK OCLs and one laboratory (Public Analyst Scientific Services) indicated an interest in participating. The samples were dispatched in May 2019 and the results were reported by the deadline of 15 June 2019.

Three samples (approximately 25g each) were received:

- Poultry compound feed containing diclazuril (authorised level)
- Poultry compound feed that may contain diclazuril (cross-contamination level)
- Poultry compound feed containing narasin and nicarbazin (authorised levels)

The method involves solvent extraction, SPE clean-up and determination by LC-MS/MS. As no appropriate long term data was available to estimate the uncertainty of each analyte the uncertainties reported were calculated as 2 times the standard deviation of the replicate results.

The z-scores obtained, all of which were satisfactory, i.e. between -2 and 2, are shown in Table 2.

Analyte	Matrix	Assigned value (mg/kg)*	Result (mg/kg)*	Z-score
Diclazuril	Authorised level	0.990	1.01	0.1
Diclazuril	Cross-contamination level	0.0092	0.007	-1.2
Narasin	Authorised level	36.6	41.44	0.9
Nicarbazin	Authorised level	37.1	42.23	0.7

*Assigned value and result in mg/kg relative to a moisture content of 12 %

Table 2: Z-scores for coccidiostat PT

Figures 5 to 8 show the results and associated uncertainties for all participants in the PT round. The uncertainties reported by LGC are based on the standard deviation of the replicate analyses of the PT samples. The identifier for LGC is C-24.

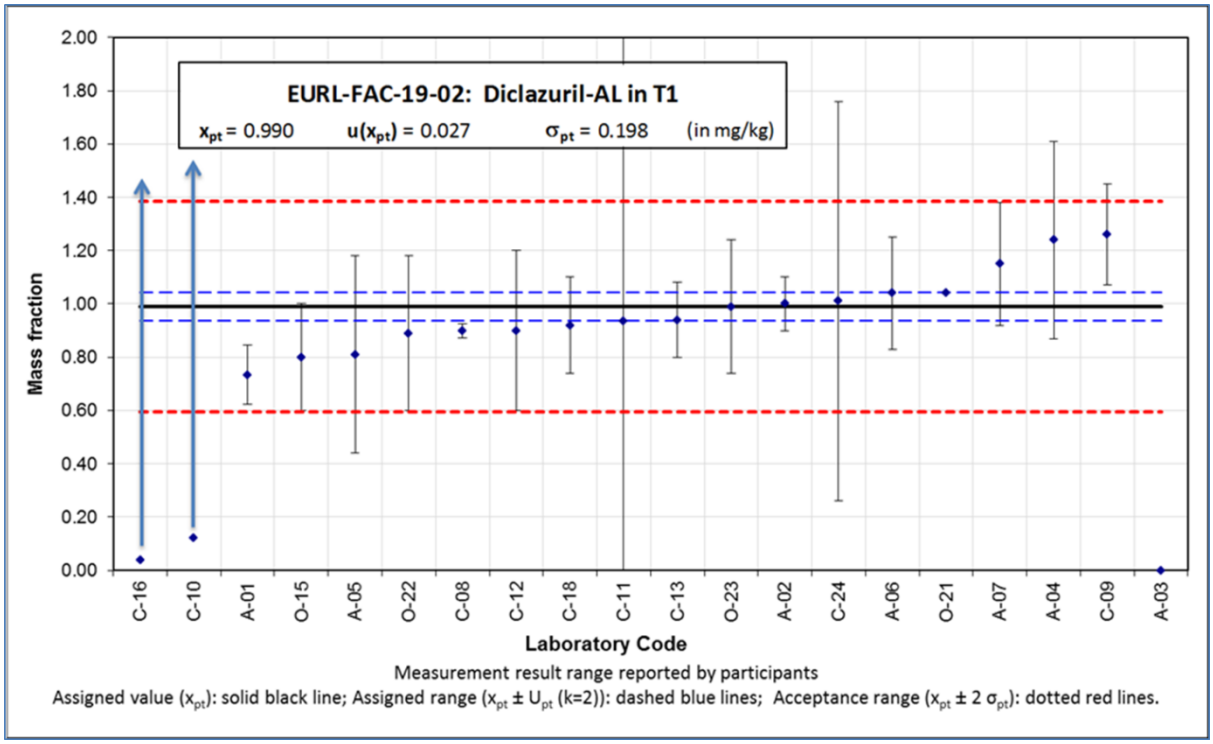

Figure 5: Summary of z-scores obtained by all participants for diclazuril in poultry feed (authorised level)

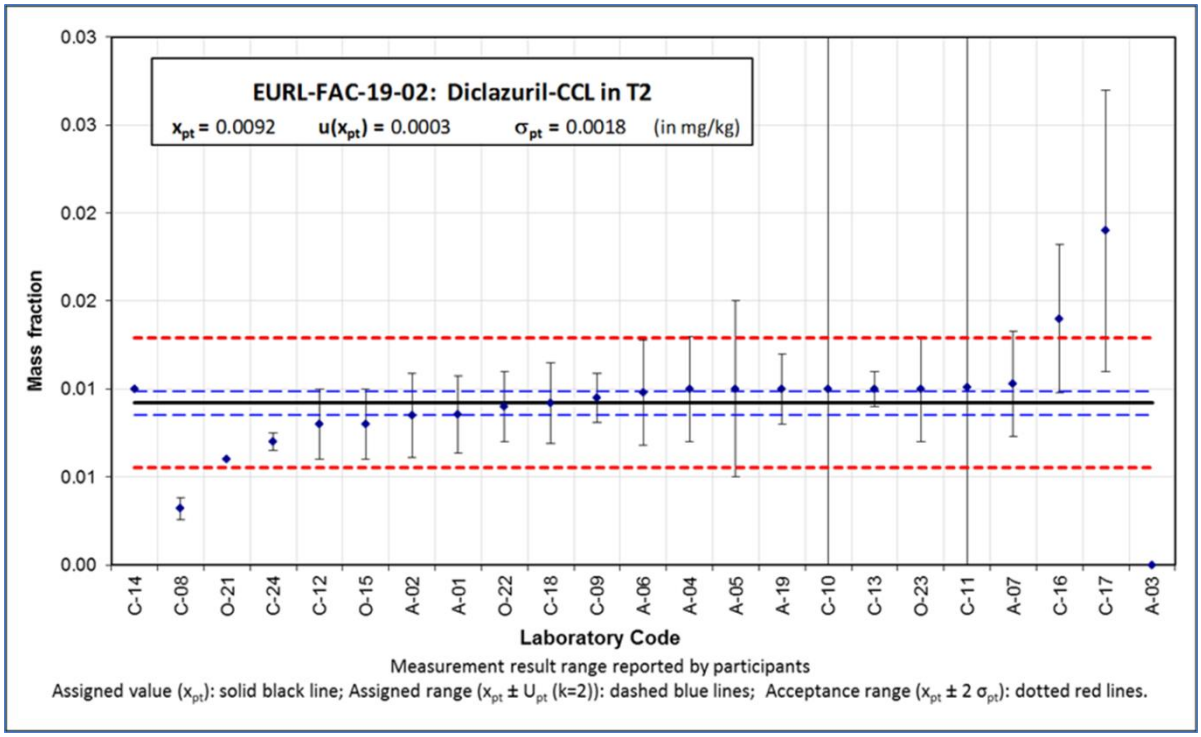

Figure 6: Summary of z-scores obtained by all participants for diclazuril in poultry feed (cross contamination level)

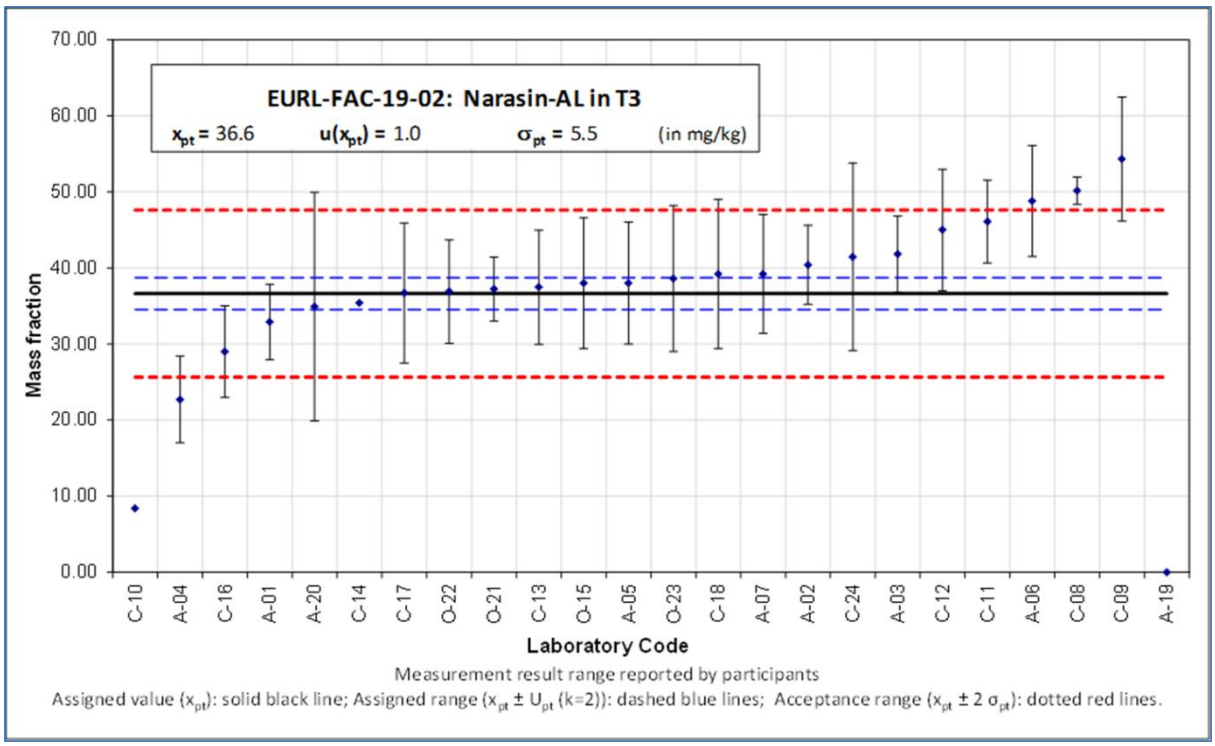

Figure 7: Summary of z-scores obtained by all participants for narasin in poultry feed (authorised level)

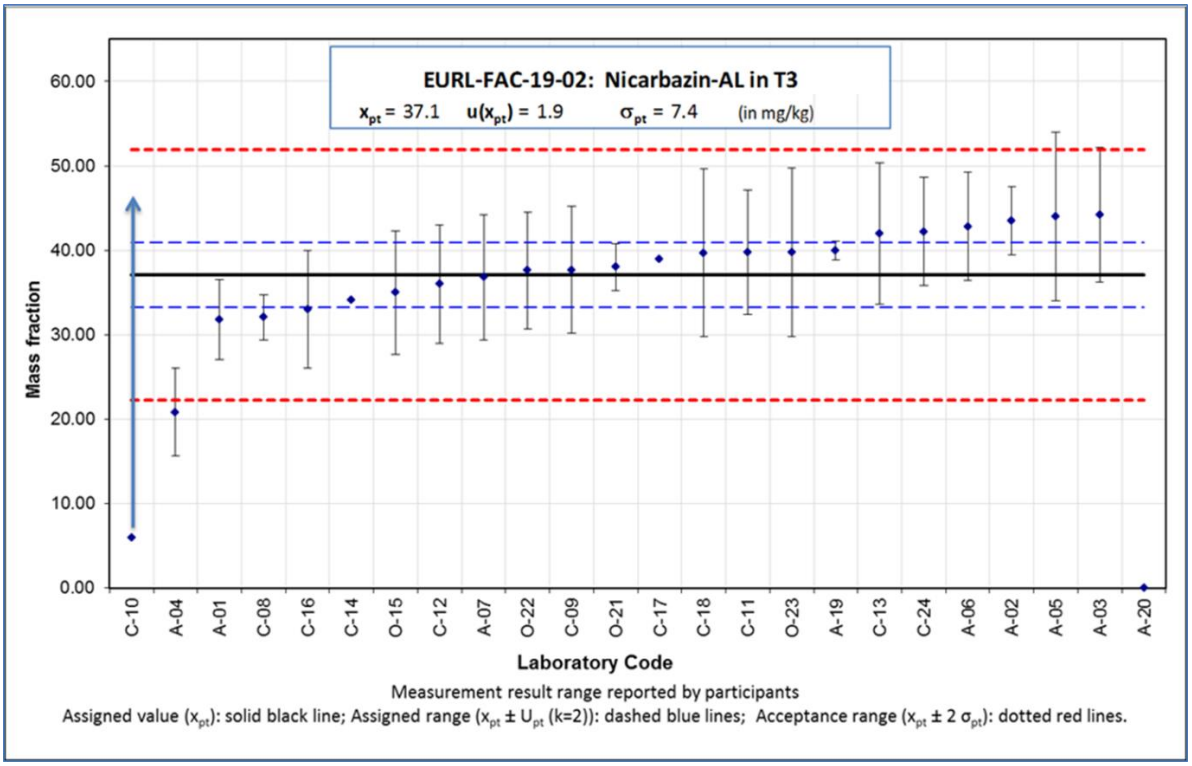

Figure 8: Summary of z-scores obtained by all participants for nicarbazin in poultry feed (authorised level)

The invitation to participate in this PT was open to OCLs and one UK lab registered to take part. The z-scores obtained by Public Analyst Scientific Services are presented in Table 3.

Analyte	Matrix	Z-score
Diclazuril	Authorised level	0.0
Diclazuril	Cross-contamination level	0.4
Narasin	Authorised level	0.4
Nicarbazin	Authorised level	0.4

Table 3: Z-scores obtained by Public Analyst Scientific Services for coccidiostat PT

The full report for the proficiency test is not currently available on the European Commission website (https://ec.europa.eu/jrc/en/publications-list) (accessed 8 April 2020).

4. EURL Proficiency Tests 2020

The EURL's aim was to carry out one PT for the determination of vitamin D_3 in 2020. Originally it was planned that the samples of poultry feed would be dispatched in the April – May period but the PT has been postponed due to the Covid-19 pandemic. An invitation to participate has been received, with the date of sample dispatch of June – July 2020, however it was explained that this may change. At the time of writing this report confirmation was awaited from the FSA as to whether it was appropriate for the UK to register to participate in the PT.

5. Urea

Discussions have been held throughout Europe recently as to whether the Regulation method for the determination of urea is applicable to certain feed types. Urea (carbamide) as a feed additive is only authorised in feed for ruminants (Commission Implementing Regulation (EU) No 839/2012). However the official method for the determination of urea in Commission Regulation (EC) No 152/2009 gives the scope as the 'method makes it possible to determine the level of urea in feed'. The principle of the Regulation method is the sample is suspended in water with a clarifying agent. The suspension is filtered. The urea content of the filtrate is determined after the addition of 4-dimethylaminobenzaldehyde (4-DMAB) by measuring the absorbance at a wavelength of 420 nm. No validation or precision data is given in the Regulation method.

At the 2018 EURL feed additives control workshop two NRLs reported that the official Regulation method was not fit for purpose for the determination of urea in pet food. Alternative methods, for example enzymatic and LC-MS/MS are apparently being used by some NRLs and official control laboratories however full validation of these methods is not available.

For these reasons towards the end of 2019 the EURL organised two trials involving the determination of urea. The first trial was a proficiency test involving the analysis of 3 samples of non-ruminant complete feed (pet feed). As one of the aims of this trial was to obtain information on the applicability of the Regulation method to the analysis of non-ruminant feed

it was decided that it would be useful for LGC to analyse the samples using two different methods (spectrophotometric method described in Regulation (EC) No 152/2009 and LC-MS/MS) to obtain a direct comparison of the two techniques in our hands. The results of the analyses are presented in Table 4.

Sample	Technique	Result reported (mg/kg)	Assigned value	Z-score
J	Spectrophotometry	4530	18.36 +/- 0.99	1228
	LC-MS/MS	17.2		-0.3
C	Spectrophotometry	3289	249.2 +/- 6.6	61
	LC-MS/MS	318		1.4
G	Spectrophotometry	4073	935 +/- 24	17
	LC-MS/MS	969		0.2

Table 4: Results from urea in pet feed trial

A significant difference can be seen between the results of the two different techniques with the results from the Regulation method (spectrophotometry) being considerably higher implying an interference when this matrix is analysed.

Tables 5 to 7 are summaries of results taken from the EURLs preliminary report. The results produced by LGC using the spectrophotometry method described in section D, annex III of Regulation (EC) No 152/2009 are labelled L08 and the results produced by LGC using LC-MS/MS are labelled L011.

1. Results for urea (@12 % moisture) in material Code J

Assigned range: x_{pt} = 18.36 ± 0.99 $U(x_{pt}, k = 2)$; σ_{pt} = 3.67 (all values are in mg/kg)

Lab Code	X_{lab}	U_{lab}	k	Technique [a]	u_{lab}	z score [b]	ζ score [b]	uncert. [c]
L001	< 1500			Gravimetry				
L002	2474	1089	2	Spectrophotometry	544.5	669	5	c
L003	2212	111	2	Spectrophotometry	55.5	597	40	b
L004	2667			Spectrophotometry	0	721	5349	b
L005	5800.2	1160	2	Spectrophotometry	580.0	1574	10	a
L006	3812.09	1143.63	2	HPLC-UV	571.815	1033	7	a
L007	1400	70	2	Spectrophotometry	35.0	376	39	b
L008	4530	466	2	Spectrophotometry	233.0	1228	19	a
L009	6167	185	2	Spectrophotometry	92.5	1674	66	b
L010	3549	890	2	Spectrophotometry	445.0	961	8	a
L011	17.3	2	2	LC-MS/MS	1.0	-0.3	-1.0	a
L012	< 50	~~15~~	~~2~~	LC-MS/MS	7.5			
L013	< 100			LC-MS/MS				
L014	3494.92	327.42	2	Spectrophotometry	163.710	947	21	a
L015	4054.87			Spectrophotometry	0	1099	8153	b
L016	41	17	1	LC-MS/MS	17.0	6	1.3	c
L017	2725	2000	2	Spectrophotometry	1000.0	737	2.7	c
L018	< 1500			Enzymatic				
L019	20.5	4.4	2	LC-MS/MS	2.20	0.6	0.9	a
L020	< 50			LC-MS/MS				
L021	148	40	2	Enzymatic	20.0	35	6	a
L022	< 1700			Enzymatic				
L023	224			Enzymatic	0	56	415	b
L024	3660	681	2	Spectrophotometry	340.5	992	11	a
L025	2323	697	2	Spectrophotometry	348.5	628	7	a
L026	331.52	48.9	2	HPLC-UV	24.45	85	13	a
L027	68.8	53.6	2	In-house photometric	26.80	14	1.9	c
L028	< 10			LC-MS/MS				
L029	680	366	2	Enzymatic	183.0	180	3.6	c
L030	1304	538	2	Spectrophotometry	269.0	350	5	c
L031	< 500	130	2	Enzymatic	65.0			

[a] technique used to determine urea in the sample "as is"

[b] Performance: satisfactory, questionable, unsatisfactory,

[c] a: $u_{min}\,(u(X_{pt})) \leq u_{lab} \leq u_{max}(\sigma_{pt})$; b: $u_{lab} < u_{min}$; and c: $u_{lab} > u_{max}$

Table 5: Summary of results for urea PT (sample J)

2. Results for urea (@12 % moisture) in material Code C

Assigned range: x_{pt} = 249.2 ± 6.6 $U(x_{pt}, k = 2)$; σ_{pt} = 49.8 (all values are in mg/kg)

Lab Code	X_{lab}	U_{lab}	k	Technique [a]	u_{lab}	z score [b]	ζ score [b]	uncert. [c]
L001	< 1500			Gravimetry				
L002	< 2450			Spectrophotometry				
L003	1440	77	2	Spectrophotometry	38.5	24	31	a
L004	2257			Spectrophotometry	0	40	611	b
L005	4897.8	979.6	2	Spectrophotometry	489.80	93	9	a
L006	4468.33	1340.5	2	HPLC-UV	670.25	85	6	a
L007	1148	57	2	Spectrophotometry	28.5	18	31	a
L008	3289	1559	2	Spectrophotometry	779.5	61	3.9	c
L009	2364	71	2	Spectrophotometry	35.5	42	59	a
L010	2222	630	2	Spectrophotometry	315.0	40	6	a
L011	318	108	2	LC-MS/MS	54.0	1.4	1.3	a
L012	252	50	2	LC-MS/MS	25.0	0.1	0.1	a
L013	300	22.6	2	LC-MS/MS	11.30	1.0	4	a
L014	2182.73	219.48	2	Spectrophotometry	109.740	39	18	a
L015	4419.1			Spectrophotometry	0	84	1269	b
L016	57	24	1	LC-MS/MS	24.0	-3.9	-8	c
L017	< 2400			Spectrophotometry				
L018	< 1500			Enzymatic				
L019	251	53	2	LC-MS/MS	26.5	0.0	0.1	a
L020	260	52	2	LC-MS/MS	26.0	0.2	0.4	a
L021	258	40	2	Enzymatic	20.0	0.2	0.4	a
L022	< 1700			Enzymatic				
L023	2824			Enzymatic	0	52	783	b
L024	3480	640	2	Spectrophotometry	320.0	65	10	a
L025	980	294	2	Spectrophotometry	147.0	15	5	a
L026	469.96	36.3	2	HPLC-UV	18.15	4.4	12	a
L027	257.7	28.4	2	in-house photometric	14.20	0.2	0.6	a
L028	1249	125	2	LC-MS/MS	62.5	20	16	a
L029	824	98	2	Enzymatic	49.0	12	12	a
L030	975.4	536	2	Spectrophotometry	268.0	15	2.7	c
L031	659	130	2	Enzymatic	65.0	8	6	a

[a] technique used to determine urea in the sample "as is"

[b] Performance: satisfactory, questionable, unsatisfactory,

[c] a: $u_{min}(u(X_{pt})) \leq u_{lab} \leq u_{max}(\sigma_{pt})$; b: $u_{lab} < u_{min}$; and c: $u_{lab} > u_{max}$

Table 6: Summary of results for urea PT (sample C)

3. Results for urea (@12 % moisture) in material Code G

Assigned range: x_{pt} = 935 ± 24 $U(x_{pt}, k = 2)$; σ_{pt} = 187 (all values are in mg/kg)

Lab Code	X_{lab}	U_{lab}	k	Technique [a]	u_{lab}	z score [b]	ζ score [b]	uncert. [c]
L001	< 1500			Gravimetry				
L002	2662	1171	2	Spectrophotometry	585.5	9	2.9	c
L003	2231	112	2	Spectrophotometry	56.0	7	23	a
L004	2588			Spectrophotometry	0	9	139	b
L005	4850.7	970.1	2	Spectrophotometry	485.1	21	8	a
L006	5168.67	1550.6	2	HPLC-UV	775.30	23	5	a
L007	1846	92	2	Spectrophotometry	46.0	5	19	a
L008	4073	1351	2	Spectrophotometry	675.5	17	5	a
L009	3682	110	2	Spectrophotometry	55.0	15	49	a
L010	2945	740	2	Spectrophotometry	370.0	11	5	a
L011	969	46	2	LC-MS/MS	23.0	0.2	1.3	a
L012	926	185	2	LC-MS/MS	92.5	0.0	-0.1	a
L013	1300	22.6	2	LC-MS/MS	11.3	2.0	22	b
L014	3747.23	347.44	2	Spectrophotometry	173.720	15	16	a
L015	6392.08			Spectrophotometry	0	29	459	b
L016	370	155	1	LC-MS/MS	155.0	-3.02	-3.6	c
L017	3105	2000	2	Spectrophotometry	1000.0	12	2.2	c
L018	< 1500			Enzymatic				
L019	989	210	2	LC-MS/MS	105.0	0.3	0.5	a
L020	890	140	2	LC-MS/MS	70.0	-0.2	-0.6	a
L021	1206	40	2	Enzymatic	20.0	1.4	12	a
L022	< 1700			Enzymatic				
L023	10320			Enzymatic	0	50	789	b
L024	3910	721	2	Spectrophotometry	360.5	16	8	a
L025	2157	647	2	Spectrophotometry	323.5	7	3.8	a
L026	996.09	58.92	2	HPLC-UV	29.460	0.3	1.9	a
L027	853.7	88.5	2	in-house photometric	44.25	-0.4	-1.8	a
L028	2693	269	2	LC-MS/MS	134.5	9	13	a
L029	938	223	2	Enzymatic	111.5	0.0	0.0	a
L030	1688	384	2	Spectrophotometry	192.0	4	3.9	a
L031	1315	260	2	Enzymatic	130.0	2.0	2.9	a

[a] technique used to determine urea in the sample "as is"

[b] Performance: satisfactory, questionable, unsatisfactory

[c] a: $u_{min}(u(X_{pt})) \leq u_{lab} \leq u_{max}(\sigma_{pt})$; b: $u_{lab} < u_{min}$; and c: $u_{lab} > u_{max}$

Table 7: Summary of results for urea PT (sample G)

For interest, the z-scores published for all participants in the EURL's preliminary report were sorted by technique and are summarised in Table 8. The technique which was used by the greatest number of participants (14) was spectrophotometry and all of the z-scores obtained were >3, and in some cases were >>3 (for sample J, assigned value 18 mg/kg, z-scores ranged from 350 to 1674, for sample C, assigned value 249 mg/kg, z-scores ranged from 15 to 93 and for sample G, assigned value 935 mg/kg, z-scores ranged from 4 to 29). From this limited data set it could, perhaps, be implied that the adverse effect this type of matrix (pet

food) has on the method described in Regulation (EC) No 152/2009 is concentration dependant. In the Regulation no working range for the method is given, however it is stated that if the concentration of urea is above 3 % a smaller weight of sample can be taken and conversely, if a low concentration of urea is expected the sample weight can be increased implying that the working range is not that limited. As a comparison, the maximum concentration of urea permitted in feed for ruminants with a functional rumen is 8800 mg/kg complete feed (Regulation (EU) No 839/2012).

Whilst fewer participants (7) used LC-MS/MS for the determination of the trial samples, approximately 70 % of the z-scores were satisfactory, i.e. <2.

	Sample	**J**		**C**		**G**	
	Assigned value	18.36 ± 0.99 mg/kg		249.2 ± 6.6 mg/kg		935 ± 24 mg/kg	
Technique		**Number**	**%**	**Number**	**%**	**Number**	**%**
All	z-scores	23	-	26	-	28	-
	z-scores ≤2	2	9	7	27	10	36
	z-scores >2<3	0	0	0	0	0	0
	z-scores >3	21	91	19	73	18	64
LC-MS/MS	z-scores	3	-	7	-	7	-
	z-scores ≤2	2	67	5	71	5	71
	z-scores >2<3	0	0	0	0	0	0
	z-scores >3	1	33	2	29	2	29
Spectrophotometry	z-scores	14	-	12	-	14	-
	z-scores ≤2	0	0	0	0	0	0
	z-scores >2<3	0	0	0	0	0	0
	z-scores >3	14	100	12	100	14	100
Enzyme	z-scores	3	-	4	-	4	-
	z-scores ≤2	0	0	1	25	3	75
	z-scores >2<3	0	0	0	0	0	0
	z-scores >3	3	100	3	75	1	25

Table 8: Z-scores from urea PT summarised by technique

The aim of the second urea trial organised by the EURL was to obtain performance data on the spectrophotometry method described in in section D, annex III of Regulation (EC) No 152/2009. Eleven samples of ruminant compound feed were received in November, analysed using the Regulation method and results submitted by the deadline of 10 January 2020. A report from the EURL is awaited.

6. Additional Proficiency Tests

In addition to the PTs organised by the EURL participation is undertaken in various other PTs covering a range of analytes and matrices to ensure competence is maintained is a wide range of techniques. As an example of a PT which relates directly to feed additives, a sample of pig

ration (FAPAS PT round 10164) was analysed for, *inter alia*, vitamin E using the method described in Regulation (EC) No 152/2009 and zinc. The z-scores obtained were -0.1 and -0.2 respectively.

In the event that, going forward, the UK is unable to participate in PT rounds organised by the EURL, alternative means of providing evidence of continuing competence will need to be found. PTs involving feed are limited so the FAPAS round for vitamin E and pig ration will be participated in again in 2020. Participation in a FAPAS round for coccidiostats and chloramphenicol in poultry feed is also under consideration.

7. Feed Additive Authorisation

The current process for authorisation of feed additives for use in the EU is summarised in Appendix 1. One step of the authorisation process involves the EURL preparing a report detailing and commenting on the methods suggested by the applicant for control of the feed additive. Methods are required for the identification / characterisation of the feed additive and determination of the active substance in the feed additive, premixtures, feedingstuffs and, when appropriate, water. The reports are then sent to the NRLs for review and comments before the report is finalised by the EURL and sent to the Commission. Examples of reports received recently include Bacillus subtilis ABS747, L-valine and sunset yellow FCF.

Article 14 of Regulation (EC) No 1831/2003 states that authorisations under that regulation shall be renewable for 10 year periods and applications for renewal shall be sent to the Commission at the latest one year before the expiry date of the authorisation. A review of the EU Register of Feed Additives showed that the authorisation of 33 feed additives expire in 2021, therefore it is expected that approval for re-authorisation will be sought in the next year, if the process has not already been started.

8. OCL Training

In the first week of April 2019 a week long residential course was held at Reading University on the Examination and Analysis of Foods. One of the sessions in the course was on feeds and feed additives with presentations given by Kevin Wardle of Public Analysts Scientific Services and Mark Bond, Food Standards Agency, respectively. Both lectures were well received. The course is organised by the Government Chemist programme and the APA Educational Trust and accredited by the RSC. The course consists of a mix of lectures, laboratory practical sessions and interactive exercises and the participants are mainly from UK Public Analyst laboratories. The course syllabus works on a two year cycle so that it is expected that a section on feed and feed additives will be included in 2021.

The residential course that was scheduled to take place at Reading University during the week commencing 30 March 2020 was unfortunately postponed due to the Covid-19 pandemic. It is hoped that the practical sessions will be held at a later date. Currently work is in progress to record many of the presentations that were to be given at reading this year so that they can be viewed online.

The UK OCLs are regularly asked if they have any current training needs as one of the roles of the NRL is to organise training exercises for OCLs. It has been explained that training provided by the NRL does not necessarily have to take the form of a formal PT but could be more specific or individual training if it would be of benefit. The OCLs have also been asked if, as an alternative to analysis, there were any areas where an advice note or guidance would

be useful, for example with the calculation and application of measurement uncertainty, interpretation of any specific legislation or application of tolerances.

At a recent Association of Public Analysts Training Committee meeting a need was expressed for feed additive training, for example on additive categorisation and labelling. Further information is awaited from Agricultural Analysts[1] on the exact nature of the training required.

9. Meetings and advice

An update of NRL activities was presented to the Food Standards Agency on 12 July 2019.

A meeting was also held with the FSA on 22 January 2020 to discuss the authorisation processes for both feed additives and GMOs following EU exit and during the transition period.

Michael Walker, Association of Public Analysts (APA) Training Officer, attended APA Training Committee meetings and gave updates on NRL activities when required.

In addition to discussions on the role of NRLs following EU exit and capability during the COVID-19 pandemic, throughout the year, advice was provided to the Food Standards Agency on the following topics:

- Methods of analysis to be used for control and by manufacturers
- Use of Food Chemical Codex method of analysis for L-threonine
- Definition and purpose of analysing crude ash.

Advice was also provided to OCLs on:

- Methods for the determination of selenium in feed
- The determination of formaldehyde in feed grade urea.

10. EURL Workshops

The 19th workshop of the EURL Feed Additives (EURL-FA) authorisation was organised and held at the Joint Research Centre in Geel on October 22-23 2019. A total of twenty nine participants, representing 17 NRLs, DG SANTE, EFSA, and the EURL-FA, took part in the workshop. The 8th annual workshop of the EURL-FA control was held at the same venue on October 23-24 2019. This workshop was attended by forty participants representing 20 NRLs (including Norway and Switzerland) and National Official Control Laboratories (OCLs) from 18 European Member States, DG SANTE and the EURL-FA. Executive summaries of the workshops can be found in Appendix 2 and Appendix 3.

LGC as the UK NRL was asked by the competent authority not to attend the workshops.

According to the minutes, highlights of the workshops included:

[1] Public Analysts are often co-appointed as Agricultural Analysts responsible for the official control analysis of feedingstuffs including feed additives.

Discussion on the results of the carotenoid proficiency test
The representative from the French NRL questioned why the average of the results reported by participants differed from the homogeneity results by around 20 %. The explanation given by the EURL was that, unlike the results reported by participants, the homogeneity results were not adjusted to 12 % moisture and this could make a difference of approximately 6 - 7 % on the mass fraction reported. Also, a non-correct integration and summing-up of all isomers related to one compound would make a difference.

Discussion on the results of the carotenoid proficiency test
A discussion was held regarding whether the results reported by NRLs had been adjusted to 12 % moisture by the EURL. It was explained by the EURL that they, as a PT provider, are not allowed to manipulate values provided by participants. It was agreed that as of 2020 the EURL will continue to request that participants report the moisture content of the samples analysed but will specify in the accompanying letter that the reported results should be adjusted to 12 % moisture by the participants.

Optiphos®[2]
The expression of results for the determination of Optiphos® was discussed including the use of a conversion factor so that the results from the ISO method can be compared to those using the Optiphos® method published by the EURL-FA.

Urea in feed
The question around the applicability of the method described in Regulation (EC) No 152/2009 to feed other than ruminant feed was described and discussed. Participation in the, then, upcoming proficiency test for the determination of urea in pet food and method validation study of the official method was encouraged.

'The truth in the bottle'
This JRC presentation was on certified reference materials (CRMs); why are they needed, how are they produced and which are available?

11. EURL Work Programme for 2020

The work programme for 2020 for the EURL Feed Additives – Control was described at the annual workshop in October. The main items were:

- Maintenance and update of the web-based methods overview.
- Helping NRLs obtaining pure standards of active substances in feed additives when not commercially available and when the standards are to be used in the frame of, and legally required, for official control.
- Organisation of a proficiency test on the determination of vitamin D_3 and antioxidants at authorised levels in feedingstuffs. A second proficiency test or training is being considered and will be announced later in the year.
- The EURL is having internal discussions on the possibility of on-site training for the correct identification of specific feed additives, for example determination of the chelate

[2] OptiPhos® is a commercially available phytase.

degree of trace elements using mid infra-red spectroscopy, or training on the 'use of reference materials and the estimation of measurement uncertainty'.

- Follow-up survey with NRL's who underperformed in the 2019 proficiency tests (carotenoids and coccidiostats). The survey will consist of a questionnaire with root-cause analysis, corrective actions, evidence and comments.
- Continuation of method development, single lab validation and a collaborative trial for the determination of p-phenetidine[3] in feedingstuffs.
- Establishment of a MS/MS semi-targeted profiling database of feed additives, initially setting up a database of MS/MS spectra for coccidiostats then extending to include at least another class of feed additives.
- Organisation of the annual workshop, with a provisional date of October 2020.

12. NRL Forward Workplan

Secretariat services
The NRL will continue to disseminate information, as appropriate, between the EURL, FSA and OCLs.

Advice and representation within the UK and internationally
Advice will be provided, as requested. Subject to CA approval, the NRL will be represented at relevant meetings, for example, the EURL workshops, for as long as the UK is invited to attend.

Production of standard operating procedures, codes of practice and guidance documents
As requested by the FSA, contribution will be made to the development of standardised operating procedures, relevant codes of practice and guidance documents for use by OCLs and other relevant laboratories.

Compliance assessment via audits and ring trials
The NRL will participate in relevant PTs and method validation studies and co-ordinate the participation of UK OCLs, as appropriate. Training exercises for the UK OCLs will be planned and organised as and when they are deemed necessary.

Co-ordination within the UK of international initiatives
Where appropriate, the recommendations of international organisations related to the standardisation of testing methods will be co-ordinated.

Communication of results and data use
The NRL will provide updates to the FSA, including an annual report.

[3] p-phenetidine, a compound found in the feed additive ethoxiquin and a possible mutagen, (see EFSA FEEDAP Panel (EFSA Panel on Additives and Products or Substances used in Animal Feed), 2015. Scientific opinion on the safety and efficacy of ethoxyquin (6-ethoxy-1,2-dihydro-2,2,4-trimethylquinoline) for all animal species. EFSA Journal 2015;13(11):4272, 58 pp. doi:10.2903/j.efsa.2015.427)

13. NRL website

Information about LGC's NRL roles can be found on our website at:

https://www.lgcgroup.com/what-we-do/national-laboratory-and-government-roles/national-laboratory-roles/national-reference-laboratories/ (accessed 18 February 2020)

14. Official Control Laboratory accreditation status

One of the NRLs roles is to maintain a list of the accreditation status of relevant OCLs. Table 9 presents the accreditation status with regards to feed and feedingstuffs as of February 2020, according to the schedules published on the UKAS website.

The major change in the accreditation scope of the OCLs since it was last reviewed in October 2018 was the addition of a flexible scope for contaminants and composition by Edinburgh Scientific Services. The scope of the accreditation covers the development and modification of methods for the analysis of feeds using generic in-house procedures for the techniques GC, GC-MS, HPLC, AAS, ICP-OES, UV/VIS spectrophotometry, microscopy and classical techniques.

Laboratory	UKAS accreditation status - Feed additives
Hampshire Scientific Services	No reference to feed / feed additives apart from melamine in feed products containing milk or soya.
Kent Scientific Services	The following are accredited in animal feeding stuffs - Aflatoxins B1, B2, G1 and G2, Ash Content, Citrinin, Crude Fibre, Copper, Inorganic Arsenic, Lead, Cadmium, Moisture, Nitrogen, Oil, Vitamin A, Vitamin E, Total Mercury, Fumonisins, B1 and B2, Ergot Alkaloids (Ergocornine, Ergocristine, Ergocryptine, Ergometrine, Ergosine, Ergotamine), Deoxynivalenol, Arsenic, Histamine, Mercury. The following are accredited in unspecified foods and animal feeds - Additives, colourings, preservatives and related contaminants & composition - Development and modification of methods for food and feed analysis using generic in-house methods using the techniques HPLC, LC/MS, GC, GC/MS, AAS, UV VIS, spectrophotometry, microscopy, ELISA and wet chemistry (drying, weighing and titration).
Lancashire County Scientific Services	The following are accredited in animal feeding stuffs - Ash, Crude oil and fat, Fibre, Moisture, Protein (calculated value), Vitamin A, Vitamin E, Cadmium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Zinc.
Aberdeen Scientific Services (Aberdeen City Council)	The following are accredited in animal feeding stuffs - Contaminants and composition, Aflatoxins - B1, B2, G1 and G2 and total aflatoxins, Arsenic, Selenium, Ash, Calcium, Magnesium, Iron , Copper, Manganese, Zinc, Lead, Cadmium, Mercury, Moisture, Nitrogen, Oil, Protein, Crude fibre, Vitamin A, Vitamin E.
Dundee City Council Scientific Service (Tayside Scientific Services)	The following are accredited in animal feeding stuffs - Additives, colourings, preservatives and related contaminants, Composition, Aflatoxin B1, B2, G1, G2, Ochratoxin A, Deoxynivalenol, Zearalenone, Ash, Crude fibre, Moisture, Oil (total), Nitrogen and crude protein, Vitamins A, Vitamin E, Cobalt, Copper, Iron, Manganese, Zinc, Cadmium, Lead, Arsenic, Selenium.
Edinburgh Scientific Services (The City of Edinburgh Council)	The following are accredited in animal feeding stuffs - Aflatoxins B1, B2, G1 and G2, Ochratoxin A, Ash, Acid insoluble ash, Crude fibre, Oil/fat, Moisture, Nitrogen, Protein, Arsenic, Cadmium, Cobalt, Copper, Lead, Mercury, Selenium, Zinc, Vitamin A and E, Isolation and confirmation of Salmonella spp.
Glasgow Scientific Services	The following are accredited in animal feeding stuffs - Ash, Crude fibre, Crude oils and fats, Moisture, Nitrogen, Protein, Crude protein, Cadmium, Copper, Lead, Selenium, Zinc, Calcium, Copper, Iron, Magnesium, Manganese, Phosphorus, Vitamin A, Vitamin E.
Minton, Treharne and Davies Limited	The following are accredited in animal feeding stuffs - Ash, Copper, Crude fibre, Lead, Cadmium, Moisture, Nitrogen and Protein, Oil, Generic protocol for the development of methods of analysis.
Public Analyst Scientific Services, Wolverhampton	No reference to feed / feed additives.

Table 9: Accreditation status of UK OCLs as of February 2020

15. Complimentary work

Food and feed legislation

Under the Government Chemist function, feed and food law is regularly reviewed and a report placed on the Government Chemist website describing recent changes to relevant law. The compendium of UK food and feed legislation with associated context and changes can be found at: https://www.gov.uk/government/organisations/government-chemist

Theobromine

For information, we have been working on a validated method for the determination of theobromine in feed for CEN 327 working group 5.

The protocol has been published as BS EN 17270:2019 Animal feeding stuffs: Methods of sampling and analysis - Determination of theobromine in feed materials and compound feed, including cocoa derived ingredients, by liquid chromatography.

A paper on the validation of the method which includes the results of a collaborative trial in which the UK OCLs were well represented, has been published in JAPA.
(http://www.apajournal.org.uk/html/japa_vol_47_pg__01-35.html) (accessed 9 April 2020)

Appendix 1: Summary of current authorisation process for feed additives

The procedure for authorisation of feed additives is described in Commission Regulation (EC) 1831/2003 on additives for use in animal nutrition https://eur-lex.europa.eu/legal-content/EN/TXT/?qid=1558444348834&uri=CELEX:02003R1831-20151230 (accessed 21 May 2019)

In summary, the authorisation procedure involves the following steps:

- The applicant sends a declaration form to the EURL which checks the information. The appropriate fee is calculated by the EURL and paid by the applicant.
- The completed application form is sent by the applicant to the European Commission (DG Santé).
- Reference samples are sent to the EURL by the applicant.
- The Commission (EFSA) makes all the information provided by the applicant available to the EURL which evaluates the technical dossier (see below for information provided). At the same time EFSA carry out a scientific assessment.
- The Rapporteur (EURL or a NRL) prepares an initial report which is made available to NRLs for peer review. Comments received are incorporated into a revised version of the evaluation report which is sent, via the EURL if they were not the Rapporteur, to EFSA, DG Santé and the applicant.
- If the evaluation report indicated that further testing and / or validation is required, the EURL provides a detailed work-plan.
- The Commission prepare a draft Regulation to grant or deny authorisation.

Authorisations are valid for 10 years and applications can be made to the European Commission to renew authorisation.

EFSA are required to provide an opinion within 6 months of receipt of a valid application and the Commission is required to prepare a draft Regulation to grant or deny authorisation within 3 months or receiving EFSA's report.

Guidelines to the authorisation process have been published on the EURL Feed Additives webpage: https://ec.europa.eu/jrc/en/eurl/feed-additives/guidance-for-applicants (accessed 21 May 2019)

The applicant is required to send the following information to EFSA who then forward it to the EURL for review:

(a) name and address;

(b) the identification of the feed additive, a proposal for its classification by category and functional group under Article 6 of Commission Regulation (EC) 1831/2003, and its specifications, including, where applicable, purity criteria;

(c) a description of the method of production, manufacturing and intended uses of the feed additive, of the method of analysis of the additive in feed according to its intended use and, where appropriate, of the method of analysis for the determination of the level of residues of the feed additive, or its metabolites, in food;

(d) a copy of the studies which have been carried out and any other material which is available to demonstrate that the feed additive satisfies the criteria laid down in Article 5(2) and (3), i.e. that it is safe and has the necessary favourable characteristics;

(e) proposed conditions for placing the feed additive on the market, including labelling requirements and, where appropriate, specific conditions for use and handling (including known incompatibilities), use levels in complementary feedingstuffs and animal species and categories for which the feed additive is intended;

(f) a written statement that three samples of the feed additive have been sent by the applicant directly to the Community reference laboratory;

(g) for additives which, according to the proposal under point (b), do not belong to either category (a) or category (b) referred to in Article 6(1) (technological additives and sensory additives respectively), and for additives falling within the scope of Community legislation relating to the marketing of products consisting of, containing or produced from GMOs, a proposal for post-market monitoring;

(h) a summary containing the information provided under points (a) to (g);

(i) for additives falling within the scope of Community legislation relating to the marketing of products consisting of, containing or produced from GMOs, details of any authorisation granted in accordance with the applicable legislation.

The fees for the authorisation of a new feed additive are detailed in Commission Regulation (EC) No 378/2005 (https://eur-lex.europa.eu/legal-content/EN/TXT/?qid=1558444527803&uri=CELEX:02005R0378-20151022) (accessed 21 May 2019) and are EUR 6000 for one feed additive.

According to the EURL for feed additives authorisation's activity report for 2017, 31 applications were evaluated during the year.

Appendix 2: Executive summary of the proceedings of the Feed Additives Authorisation workshop

Executive Summary

The 19th workshop (WS) of the EURL Feed Additives (EURL-FA) authorisation was organised and held in Geel on October 22-23, 2019. A total of twenty nine participants, representing 17 National Reference Laboratories (NRLs), DG SANTE, EFSA, and the EURL-FA, took part in the workshop. This year, following the request of the NRLs, the workshop also included half day training on the evaluation of a feed additive dossier.

C. von Holst, operating manager of the EURL-FA, started the event by introducing the agenda of the workshop to the participants followed by a presentation dealing with the main aspects to take into consideration when evaluating feed additive dossiers.

The participants were then divided in four groups where one different case scenario was used as the base of the training. Four different case scenarios were selected, namely amino acids, chelate trace elements, enzymes and mycotoxin binders. Each of the group was chaired by one member of the EURL-FA. Due to time constraints the training was restricted to find the key information and the peculiarities for each of the selected case scenarios in order to complete an ad-hoc check list created for this purpose.

Z. Ezerskis (EURL-FA) gave the presentation on EURL-FA authorisation activities and deliverables of 2019 such as management of declaration forms and feed additive samples, evaluation of dossiers and drafting evaluation reports, the progress of the project related to the publishing on the EURL website the recommended analytical methods for official control of feed additives and the finalised annual report on EURL activities in 2018.

The presentation given by A. Rodriguez (DG SANTE) informed about the status of the authorisation of vitamin B_2: additives re-evaluated, additives to be withdrawn from the market after the re-evaluation and the additive for which the authorisation was denied. In addition, the presentation showed how it is possible to know the status of different additives in the Register of Feed Additives published in DG SANTE web.

M.J. González de la Huebra (EURL-FA) presented the last news regarding the authorisation of Optiphos. The deadline for requesting the renewal of the authorisation of this feed additive is approaching, and the evaluation of a new 6-phytase based feed additive from the same Applicant and with phytase activities expressed in FTU units is currently ongoing. Taking into account the current scenario the near-future of Optiphos is uncertain and thus the development of an implementing strategy for the conversion factor is not considered a priority for the EURL.

The presentation from M. Innocenti (EFSA) informed about the lifecycle of an application in EFSA and some general information about the assessment process. In addition, the presentation also contained an update of the work done by the FEEDAP Panel on the dossiers in 2019 and some previsions on the workload for 2020.

Finally, P. Corbisier (JRC-Geel) presented the PCR methodology used at the JRC Geel to verify the presence of recombinant DNA (recDNA) in vitamin B_2 official control samples. The strengths and limitations of PCR methods were discussed. The EFSA guidance document and the JRC procedure available to prove the absence of recDNA in a sample (so-called zero tolerance) were compared and their different goals highlighted. The experimental verification of the compliance of an additive obtained by fermentation requires the availability of a PCR detection method able to identify the production strain as well as a

positive control for each additive. The availability of such information and materials was not foreseen in Commission Regulation (EC) No 429/2008.

The overall feedback from participants as shown in the Summary Evaluation Tables (next pages), was very positive for both parts of the workshop i.e. training and presentations. The feedback on the relevance of the program of the workshop and on the balance between formal presentations and discussions was very well perceived.

Appendix 3: Executive summary of the proceedings of the Feed Additives Control workshop

Executive Summary

The 8th Annual Workshop of the European Union Reference Laboratory for Feed Additives Control (EURL-FA Control) and the consortium of National Reference Laboratories (NRLs) was held at the Joint Research Centre in Geel on October 23-24, 2019. The event was attended by forty participants representing 20 National Reference Laboratories (NRLs) (including Norway and Switzerland) and National Official Control Laboratories (OCLs) from 18 European Member States, DG SANTE and the EURL-FA.
This 8th workshop was on one side the concluding event for the organisation of the two PT exercises conducted by the EURL-FA Control for (i) the determination of the mass fraction of authorised carotenoids in compound feed for poultry and fish and (ii) the determination of the mass fractions of diclazuril, narasin and nicarbazin in poultry compound feed. The results from the PT exercises were presented to all participants and opened to discussion. One interesting aspect was the follow-up and assessment of the evolution of the laboratories' proficiency since these 2 exercises addressed analytes tested in other PTs in previous years.
In addition the annual workshop also gave the opportunity to exchange with the NRLs on activities carried out in the field of analysis of feed additives and the issues encountered, to get an update from DG SANTE on specific issues such as the on-going revision of Commission Regulation (EC) 152/2009 or on current developments of feed additives and undesirable substances legislation. Practical information on how to apply the conversion factor previously set as an outcome of a collaborative trial organised by the EURL for the determination of a specific enzyme was given by one NRL.
The NRLs were also updated on the outcome of the tests and developments performed for the determination of urea both as an additive in ruminant feed and as undesirable substance in non-ruminant feed and encouraged to participate into the related intercomparisons. Following these studies and based on the outcome, decision regarding the revision of the Commission Regulation (EC) 152/2009 will be taken by DG SANTE.
The EURL-FA Control also informed all participating NRLs of the recent publication of the multi-analyte method it developed for the determination of the 11 authorised coccidiostats at additive and cross-contamination level and of 6 antibiotics at sub-additive levels in feed, as an EN standard.
Finally, the other activities performed by the EURL-FA Control in 2019 were reviewed and the work program for 2020 was presented. The NRLs were informed that in 2020 one PT will be organised on the determination of vitamin D3 in feed; the second foreseen PT on the determination of antioxidants in feed will be replaced by a method validation study on which the NRLs will be informed at a later stage.
The mandatory follow-up for underperforming laboratories during the 2019 PT exercises on cobalt determination will also be carried out.

Lastly the participants were encouraged to give feedback to the EURL-FA Control regarding their needs and priority list, for PTs for the period 2021 and beyond and training for the period 2020-2021. The feedback could be given at the workshop but also by electronic mail or using the dedicated web platform for discussion and exchange CIRCA-BC.

A satisfaction survey on the organisation of the PTs and of the annual workshop was conducted. The response rate for the complete survey was 71 %. The overall satisfaction rate was of 94 % and 79 % of the answers were above 85 % of satisfaction. The overall satisfaction rate for the 2019 PT on carotenoids determination was 92 % with 92 % of these positive answers being above 85 % of satisfaction. For the 2019 PT on coccidiostats, these rates were 95 % and 95 %, respectively. Finally, the results were also very good for the organisation and structure of the workshop with a 95 % satisfaction rate and 95 % above 85 % of satisfaction.